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14. ABSTRACT

A combined experimental and theoretical program is employed to address the behavior of cracks near interfaces with an emphasis on examining the relation between the T-stress term and crack path stability. Several activities exist on the theoretical and experimental fronts; examples of these are discussed below.

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a. REPORT	b. ABSTRACT	c. THIS PAGE			Ivar Reimanis
UU	UU	UU	UU		19b. TELEPHONE NUMBER 303-273-3549

Report Title

Final Progress Report

ABSTRACT

A combined experimental and theoretical program is employed to address the behavior of cracks near interfaces with an emphasis on examining the relation between the T-stress term and crack path stability. Several activities exist on the theoretical and experimental fronts; examples of these are discussed below.

Enter List of papers submitted or published that acknowledge ARO support from the start of the project to the date of this printing. List the papers, including journal references, in the following categories:

(a) Papers published in peer-reviewed journals (N/A for none)

Received		Paper
03/26/2013	3.00	Marc Rubat du Merac, Ivar E. Reimanis, Charlene Smith, Hans-Joachim Kleebe, Mathis M. Müller. Effect of Impurities and LiF Additive in Hot-Pressed Transparent Magnesium Aluminate Spinel, International Journal of Applied Ceramic Technology, (08 2012): 1. doi: 10.1111/j.1744-7402.2012.02828.x
03/28/2013	2.00	Elmabrouk, B., Berger, J. R., Phan, A.-V., and Gray, L. J. Apparent Stiffness Tensors for Porous Solids Using Symmetric Galerkin Boundary Elements, Computational Mechanics, (01 2012): 411. doi:
03/28/2013	6.00	B. Elmabrouk, J. R. Berger. Boundary element analysis for effective stiffness tensors: effect of fabric tensor determination method, Computational Mechanics, (07 2012): 0. doi: 10.1007/s00466-012-0753-3
08/26/2011	1.00	John R. Berger. Fabric Tensor Based Boundary Element Analysis of Porous Solids, Engineering Analysis with Boundary Elements, (03 2011): 430. doi:

TOTAL: **4**

Number of Papers published in peer-reviewed journals:

(b) Papers published in non-peer-reviewed journals (N/A for none)

Received Paper

TOTAL:

Number of Papers published in non peer-reviewed journals:

(c) Presentations

Invited talk, "Incorporation of microstructural effects in Green's functions and boundary element computations," National Science Foundation Workshop on the Emerging Applications and Future Directions of the Boundary Element Method, September 1-3, 2010, Akron, Ohio, USA.

M. Adam, J. R. Berger, A.-V. Phan, and I. Reimanis, "Crack Extension Near an Auxetic Particle using Symmetric Galerkin Boundary Elements," International Association of Boundary Element Methods 2011 Conference, Brescia, Italy.

B. Elmabrouk, J. R. Berger, A.-V. Phan, and L. J. Gray, "Effective Elastic Stiffness Tensors for Porous Solids with Symmetric Galerkin Boundary Element Analysis," International Association of Boundary Element Methods 2011 Conference, Brescia, Italy.

M. Rubat-du Merac and I. E. Reimanis, "Opto-electronic Properties of MgAl2O4 Spinel", 3rd Annual Colorado Center for Advanced Ceramics Conference, August 18 & 19, 2011, Estes Park, CO USA

M. Rubat du Merac, I. Reimanis, H. J. Kleebe and C. Smith, "The Role of Point Defects on the Sintering and Optical Properties of Transparent MgAl2O4 Hot Pressed with LiF", presented at the International Conference on Advanced Ceramics and Composites at Daytona Beach, January 2012.

M. Adam, J. R. Berger, A.-V. Phan, and I. Reimanis "Crack Extension Near an Auxetic Particle using Symmetric Galerkin Boundary Elements" presented at the IABEM 2011

"Effective Elastic Stiffness Tensors for Porous Solids with Symmetric Galerkin BEM,: Elmabourk, B., Berger, J. R., Phan, A.-V., and Gray, L. J., International Association for Boundary Element Methods 2011, Brescia, Italy.

Boundary Element Analysis for Effective Elastic Constants in Porous Ceramics, Elmabrouk, B. and Berger, J. R., ASCE Engineering Mechanics Conference, Univ. of Notre Dame, June 2012.

Crack Extension Near an Auxetic Particle using Symmetric Galerkin Boundary Elements, Berger, J. R., Adam, M. A., and Reimanis, I., BEM/MRM 2013, New Forest, UK, June 2013

Number of Presentations: 9.00

Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Non Peer-Reviewed Conference Proceeding publications (other than abstracts):

Peer-Reviewed Conference Proceeding publications (other than abstracts):

Received Paper

TOTAL:

Number of Peer-Reviewed Conference Proceeding publications (other than abstracts):

(d) Manuscripts

Received		Paper
02/15/2014	10.00	M. Rubat du Merac, H.-J. Kleebe, M. M. Muller, and I. E. Reimanis. Fifty Years of Research and Development Coming to Fruition; Unraveling the Complex Interactions during Processing of Tranpsparent Aluminate (MgAl2O4) Spinel, Journal of the American Ceramic Society (09 2013)
03/27/2013	5.00	. Fabric tensor, based boundary element analysis of porous solids, Engineering Analysis with Boundary Elements (06 2010)
03/28/2013	7.00	M. M. Adam, J. R. Berger, P. A. Martin. Singularities in auxetic elastic bimaterials, Mechanics Research Communications (10 2012)
03/28/2013	9.00	M. M. Adam, J. R. Berger, A. -V. Phan, I. E. Reimanis. Influence of Auxetic Particles on Crack Paths, Engineering Analysis with Boundary Elements (01 2013)

TOTAL: **4**

Number of Manuscripts:

Books

Received Paper

TOTAL:

Patents Submitted

Ivan A. Cornejo and Ivar E. Reimanis, "Alumina-Rich Glasses and Methods for Making the Same" US Provisional Patent No. 61/825,817 filed May 21, 2013.

Patents Awarded

Awards

Graduate Students

NAME	PERCENT SUPPORTED	Discipline
John Sherman	1.00	
John Mosely	1.00	
FTE Equivalent:	**2.00**	
Total Number:	**2**	

Names of Post Doctorates

NAME	PERCENT SUPPORTED
FTE Equivalent:	
Total Number:	

Names of Faculty Supported

NAME	PERCENT SUPPORTED	National Academy Member
John Berger	1.00	
Ivar Reimanis	1.00	
FTE Equivalent:	**2.00**	
Total Number:	**2**	

Names of Under Graduate students supported

NAME	PERCENT SUPPORTED	Discipline
Andrew Harper	1.00	Materials Science and Engineering
FTE Equivalent:	**1.00**	
Total Number:	**1**	

Student Metrics

This section only applies to graduating undergraduates supported by this agreement in this reporting period

The number of undergraduates funded by this agreement who graduated during this period: 0.00

The number of undergraduates funded by this agreement who graduated during this period with a degree in science, mathematics, engineering, or technology fields:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and will continue to pursue a graduate or Ph.D. degree in science, mathematics, engineering, or technology fields:...... 0.00

Number of graduating undergraduates who achieved a 3.5 GPA to 4.0 (4.0 max scale):...... 0.00

Number of graduating undergraduates funded by a DoD funded Center of Excellence grant for Education, Research and Engineering:...... 0.00

The number of undergraduates funded by your agreement who graduated during this period and intend to work for the Department of Defense 0.00

The number of undergraduates funded by your agreement who graduated during this period and will receive scholarships or fellowships for further studies in science, mathematics, engineering or technology fields:...... 0.00

Names of Personnel receiving masters degrees

NAME

Total Number:

Names of personnel receiving PHDs

NAME

Total Number:

Names of other research staff

NAME	PERCENT_SUPPORTED

FTE Equivalent:
Total Number:

Sub Contractors (DD882)

Inventions (DD882)

Scientific Progress

See attachment

Technology Transfer

Final Progress Report ARO December 15, 2013

A combined experimental and theoretical program is employed to address the behavior of cracks near interfaces with an emphasis on examining the relation between the T-stress term and crack path stability. Several activities exist on the theoretical and experimental fronts; examples of these are discussed below.

THEORETICAL DEVELOPMENT

Crack Modeling Efforts

The fracture problem illustrated in Figure 1 was examined with the boundary element method. Figure 2 shows the loading boundary conditions; these were chosen so that experiments could be performed. Figures 3 – 8 illustrate the results. Details are reported in the submitted manuscript (Influence of Auxetic Particles on Crack Paths; M. M. Adam, J. R. Berger, A.-V. Phan and I. Reimanis), but are briefly described here. Crack extension can be dramatically different near an auxetic particle when compared to extension behavior near a non-auxetic particle. For values of E_p/E_m =2, 4, it was shown that the crack was attracted to the particle when ν_p/ν_m = -1, yet when ν_p/ν_m = 1, the crack is deflected away from the particle. It was also found that when E_p/E_m = 8, 16, the crack was deflected away from the particle when ν_p/ν_m = ±1. This suggests that there may be strategies to pin extending matrix cracks by employing auxetic particles that tend to attract the crack. It was found that a soft particle (E_p/E_m = 0.5) will attract the crack when ν_p/ν_m = ±1, but if the particle is hard, (E_p/E_m = 16), the crack is only attracted when ν_p/ν_m = -1.

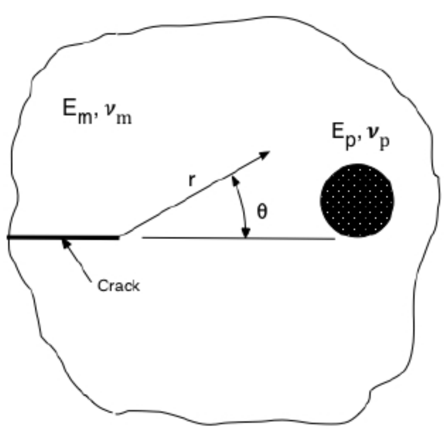

Figure 1. Crack approaching a particle.

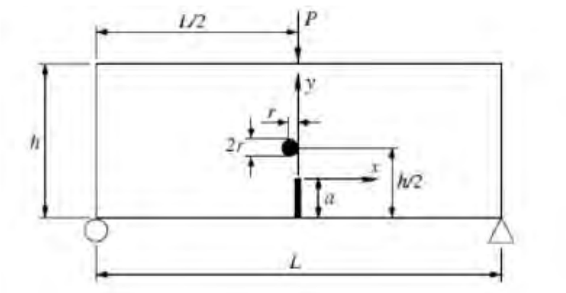

Figure 2. Crack path results when the particle Poission's ratio is negative, for the same values of Young's modulus ratio as in Figure 1. See Table 2 for corresponding values.

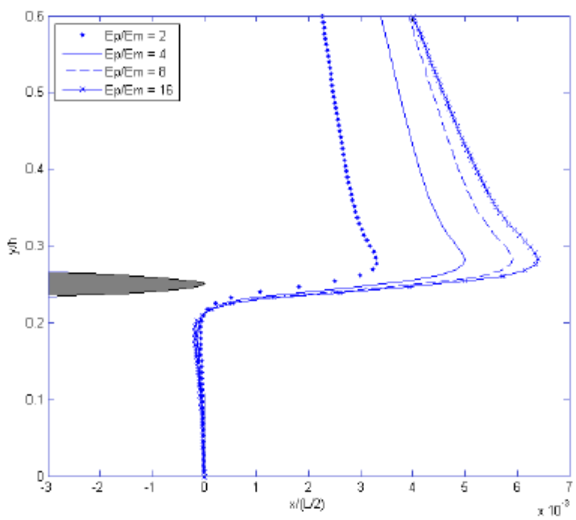

Figure 3. Comparison of results with Williams, et al. (2007) for non-auxetic particle-crack interaction, $\nu_p/\nu_m = 1$. Note that the horizontal scale is expanded to emphasize the crack path.

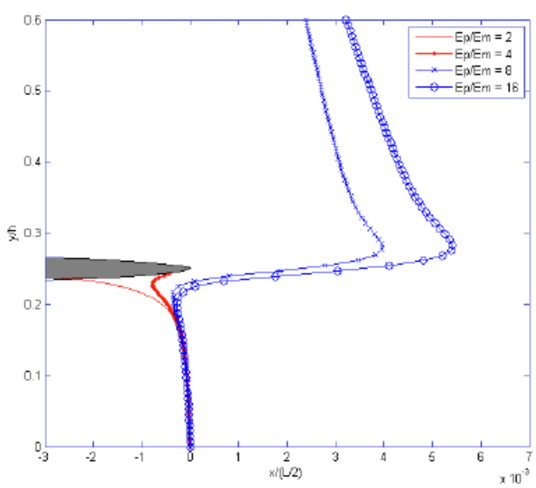

Figure 4. Crack extension near an auxetic particle, $\nu_p/\nu_m = -1$.

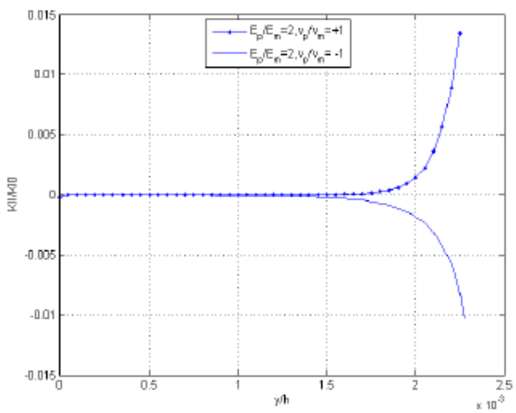

Figure 5. Normalized mode II stress intensity factor as a function of crack-tip position for $E_p/E_m = 2$, $\nu_p/\nu_m = \pm 1$.

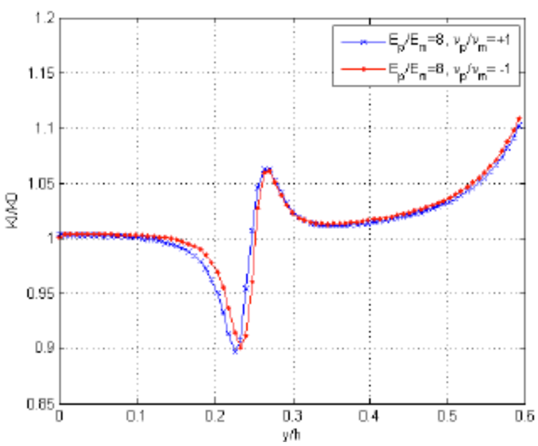

Figure 6. Normalized mode I stress intensity factor as a function of crack tip position $E_p/E_m = 8$, $\nu_p/\nu_m = \pm 1$

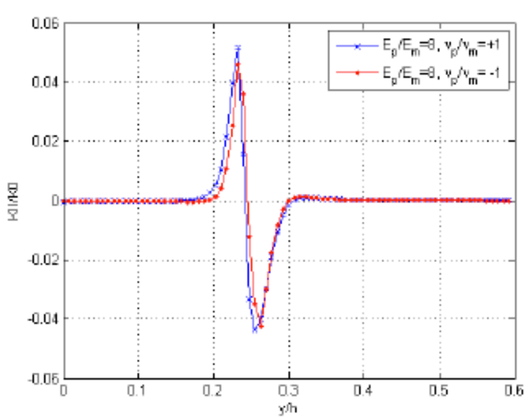

Figure 7. Normalized mode II stress intensity factor as a function of crack tip position $E_p/E_m = 8$, $\nu_p/\nu_m = \pm 1$.

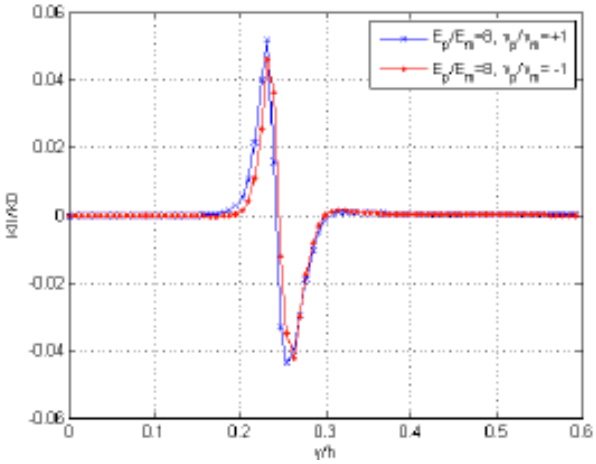

Figure 8. Crack extension near a particle, for $\nu_p/\nu_m = \pm 1$, and $E_p/E_m = 0.5$, $E_p/E_m = 2$.

EXPERIMENTAL WORK

Materials Fabrication

The effort to fabricate plates of transparent spinel in order to make layered transparent materials was continued, and progress on better understanding the sintering mechanisms for spinel was made. The motivation is to have control over sintering ultimately so that residual stress may be tailored for making layered materials.

Lanthanum niobate (LN), one of the few auxetic (negative Poisson's ratio) oxides, has been successfully synthesized. It has been very challenging to reproducibly synthesize pure LN, but we have done this via a chemical synthesis route as reported in the last progress report. Another approach was successfully applied to make LN: a mechanical ball-milling, calcination route. In this route, Nb_2O_5 and La_2O_3 (made from La_2NbO_3) powders are mixed in a 1:1 molar ratio. They are then ball milled with ZrO_2 media. Upon calcination, pure $LaNbO_4$ is formed. The mechanical aspect of ball milling is a critical part of the process. The detailed process is given below:

Mechanical Synthesis

1) Mass 8.26g of La_2NbO_3 and 6.74g Nb_2O_5 precursor powders in large weight boat
2) Combine powders in a 250ml Nalgene along with 84 balls of 1cm ZrO grinding media(from HH 375)
3) Tape up sides of the Nalgene and place on ball mill for 10 hours
4) Mass out approximately 4g of milled powder in a weight boat, and transfer to a small ceramic crucible (also found in HH 375)
5) Cover crucible with alumina lid and calcine powder in the small drop down furnace at 1200 C for 14 hours, using a 3 C/min ramp rate
6) Remove powder and grind with pestle/mortar

Figures 9 and 10 show example of the $LaNbO_4$ powders made by this process. They were shown by x-ray diffraction to be single phase. A number of experiments were performed in which the ball mill time and calcination time and temperature were varied to optimize the size of the powders.

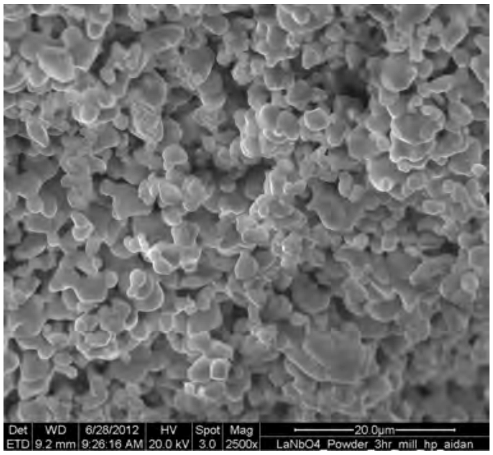

Figure 9. Scanning electron micrograph of $LaNbO_4$ powder made by mixing for 3 h and calcining at 1200 °C for 14 h.

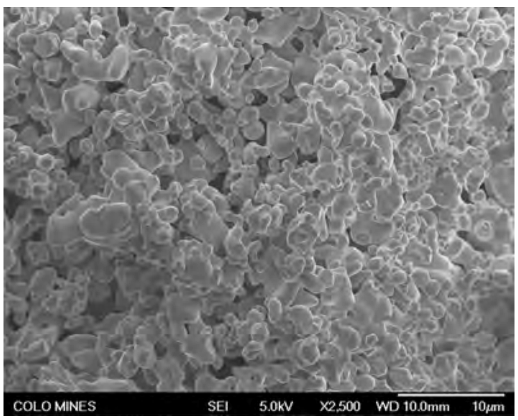

Figure 10. Scanning electron micrograph of $LaNbO_4$ powder made by mixing for 10 h and calcining at 1200 °C for 14 h.

Composites containing varying amount of LN (0 to 15 percent by volume) were made by mixing in the powder prepared above (10 h mill; 1200 °C calcination for 14 h) with purchased 10 percent by mole yttria stabilized ZrO_2 (10 YSZ Tosoh). The composites were made by cold pressing and then sintering at 1550 °C for 15 h. An example fracture surface is shown in Figure 11. Some porosity is apparent, but it appears minimal. The specimens will be cut into bars for fracture testing to establish whether or not the presence of LN significantly influence the fracture toughness.

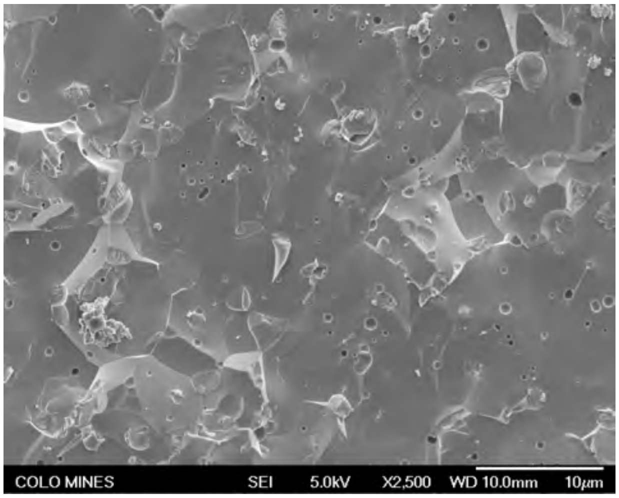

Figure 11. Scanning electron micrograph of a 15 volume percent $LaNbO_4$ – YSZ composite, made by sintering at 1550 °C for 15 h.